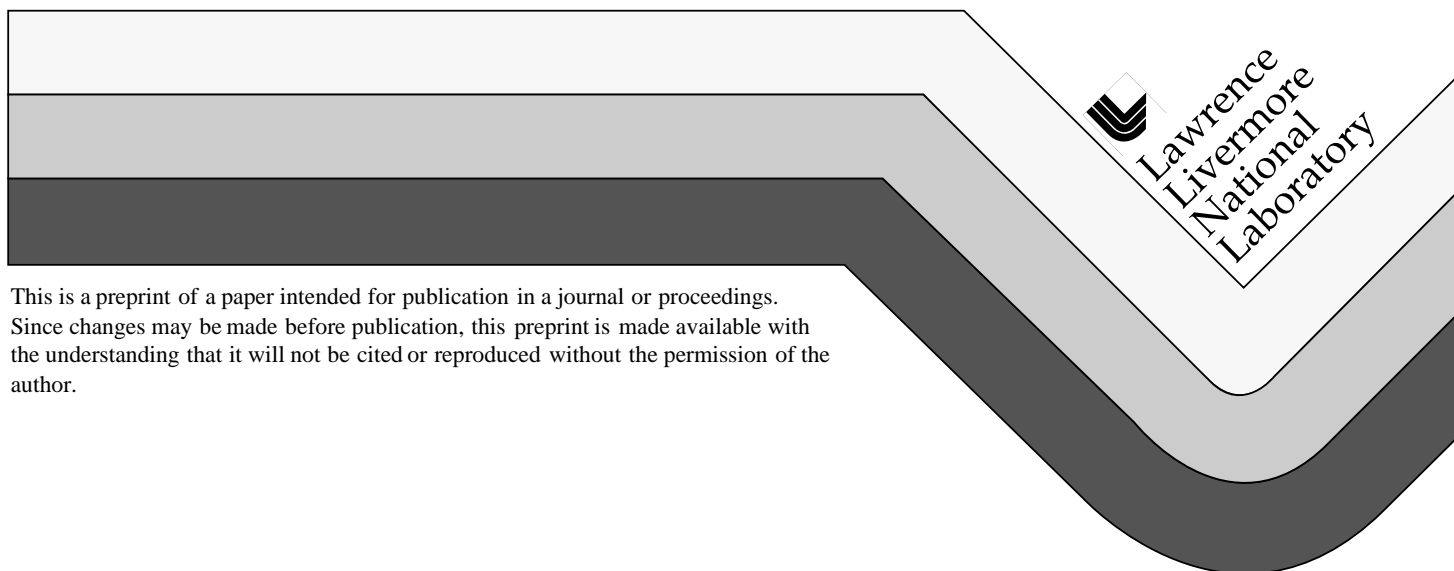


The Positron Microprobe at LLNL

W. Stoeffl
P. Asoka-Kumar
R. Howell

This paper was prepared for submittal to the
SLOPOS-8 Conference
8th International Workshop on Slow Positron Beam Techniques for Solid and Surfaces
Cape Town, South Africa
September 6-12, 1998

November 1, 1998



This is a preprint of a paper intended for publication in a journal or proceedings.
Since changes may be made before publication, this preprint is made available with
the understanding that it will not be cited or reproduced without the permission of the
author.

DISCLAIMER

This document was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor the University of California nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or the University of California. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or the University of California, and shall not be used for advertising or product endorsement purposes.

The Positron Microprobe at LLNL

W. Stoeffl, P. Asoka-Kumar, and R. Howell

Lawrence Livermore National Laboratory, Livermore, CA 94550

The electron linac based positron source at Lawrence Livermore National Laboratory (LLNL) provides the world's highest current beam of keV positrons. We are building a positron microprobe that will produce a pulsed, focused positron beam for 3-dimensional scans of defect size and concentration with sub-micron resolution. The widely spaced and intense positron packets from the tungsten moderator at the end of the 100 MeV LLNL linac are captured and trapped in a magnetic bottle. The positrons are then released in 1 ns bunches at a 20 MHz repetition rate. With a three-stage re-moderation we will compress the cm-sized original beam to a 1 micro-meter diameter final spot on the target. The buncher will compress the arrival time of positrons on the target to less than 100 ps. A detector array with up to 60 BaF₂ crystals in paired coincidence will measure the annihilation radiation with high efficiency and low background. The energy of the positrons can be varied from less than 1 keV up to 50 keV. The target can be instrumented to allow in-situ annealing, cooling and stress.

PACS: 8170C; 0780; 7870B; 6170

1. Introduction

Positron annihilation spectroscopy has emerged as a highly sensitive, nondestructive probe to study the nature, concentration, and spatial distribution of defects in materials.[1] Positrons are a unique tool to study solid state defects. Unlike electrons, positrons are repelled from most atoms and can become trapped in defects like slip-planes or voids. The change in positron lifetime and especially the change in the time-constant and amplitude of the long decay components can reveal the atomic scale defects. The microprobe will resolve changes of the defect density or defect size with high spatial resolution. By changing the implantation energy, the instrument is also sensitive to the depth distribution of defects.

For over 30 years, low-energy, monochromatic positron beams have been developed that can perform depth-resolved defect analysis of materials. The underlying physical process for converting the positrons emitted from either a radio isotope or a stopping target in an electron linac into a monochromatic beam, known as "moderation," is not very efficient. Therefore, most laboratory beams lack the brightness necessary to realize the full potential of this tool. The present status in our field is analogous to the one experienced by researchers using laboratory X-ray sources before the advent of synchrotron light sources. Creating 3-dimensional images from many thousands of individual high-statistic lifetime spectra in a pulsed positron microprobe with several re-moderation stages is only practical with an intense positron beam to begin with.

2. The high intensity positron beam

The LLNL primary source of positrons is located at the end of a 100 MeV high power linac, with a peak electron current of 400 mA and a repetition rate of 300 Hz. Each pulse lasts about 3 micro-seconds. The average linac beam power is up to 45 kW. The energetic electrons are stopped in a water-cooled tungsten target providing a shower of bremsstrahlung photons, similar to a regular X-ray machine. The pair conversion of the bremsstrahlung photons (typically 5-20 MeV) yields an intense source of energetic positrons. These positrons slow down in the tungsten converter and reach a set of well-annealed tungsten foils (25 microns thick) arranged in the form of "Venetian blinds." A fraction of the high energy positrons is slowed down to thermal energies inside the tungsten foils and is re-emitted as moderated low energy positrons. These positrons are harvested and guided through a curved 60 gauss solenoid to an experimental hall.

The 3 μ s time structure of the initial positron beam is not well suited for many experiments. Every pulse contains 3×10^7 positrons and will saturate most detection systems. Therefore, the initial beam is captured, stored, and released slowly from a penning trap to produce a time-stretched beam profile. The pulse stretcher consists of a 5 meter long 60 Gauss solenoid, a gate electrode at the entrance, a middle storage section and a barrier electrode at the exit. The voltage on the gate and barrier electrodes is higher than the primary positron energy of ~ 20 V, except for

a short interval when the gate is switched low to let in the pulse of positrons. The positron bunch is thus captured between the gate electrode and barrier electrode. After filling the trap with a positron pulse, the voltage on the trap floor is raised slowly to spill the positrons over the fixed voltage barrier. The ramp voltage is adjusted to convert the initial 3 μ s pulse into a quasi-DC beam.

The energy spread of the beam spilling over the barrier is significantly smaller than the starting beam, 4eV versus \sim 20 meV. The barrier is nominally set at 25 Volt and is followed by a 20 eV accelerator column.

Immediately following the accelerator is a two-gap time-buncher section to produce a nano second wide pulse every 50 ns containing \sim 500 positrons per pulse. A 1 GHz arbitrary waveform generator provides the non-linear sawtooth voltage for the pre-buncher.

3. Extraction of positrons from magnetic field

The microprobe will require a magnetic field-free environment for optimum performance. Therefore, the positrons have to be extracted out of the guiding magnetic field without significant degradation of the focusing phase space. This is achieved with a 5 cm diameter magnetic grid consisting of 36 tapered fins pointing toward the center similar to the “spokes” of a wheel. The high permeability stainless steel of the spider is EDM machined from a 1 cm thick block with a single continuous cut. The fins are only 50 micro-meter thick at their tips. As the spokes approach the center they are terminated at three different distances from the center to have an effective magnetic field termination and $>$ 90% transmission. Since the electric potential at the magnetic spider is typically 2000 V, the magnetic path to the solenoid return yoke has a 1 mm gap inside the vacuum tube to allow for electrical isolation. Within a few mm from the spider, the magnetic field drops to less than 0.3 Gauss.

Before the positrons pass the magnetic spider, they are accelerated to 2000 Volt to make the exit from the magnetic field as non-adiabatic as possible. Beyond the magnetic spider, the positrons are guided by electrostatic lenses only. The earth magnetic field is reduced to a few mG by a mu-metal shield around the beam tubes and the switchyard chambers.

A switchyard located 40 cm from the magnetic grid can divert the electrostatically transported beam to various experiments. The user beam lines are designed with electrostatic elements

and the beam is diverted to different end stations using more electrostatic switchyards.

4. The positron microprobe

The pulsed positron microprobe will be the central part of the high intensity positron program. The microprobe will provide 100 ps bunches of positrons with an intensity of 10^7 positrons/s. The final spot size will be about 1 μ m with a bunch repetition rate of 20 MHz. Thus the average “bunch” at the target consists of one or zero positrons. The microprobe will take positron lifetime data to determine the defect size and concentration over a spatial volume as small as $0.025 \mu\text{m}^3$. Such a small sample volume contains only about 10^{10} Atoms. The energy of the positron beam can be varied 1-50 keV to sample the near surface regions and buried features. The vacuum in the microprobe chamber and target area will be about 10^{-10} torr.

The positron beam exiting the magnetic grid of the stretcher has the following properties: diameter = 1.2 cm, half-angle of divergence $< 1^\circ$, energy = 2 keV, a pulse width = 1 ns, and a repetition rate = 20 MHz. Conversion of this beam into a microprobe is challenging and is achieved using several phase compression techniques.

The most important principle is the brightness enhancement of the beam using successive stages of moderation. Due to the limitations in the primary positron flux (unlike in an electron microscope, where there is an abundant supply of electrons), collimation is not practical for attaining a micron beam spot. The brightness of a positron beam (flux \bullet spotsize) can be increased when a beam of fast positrons (\sim 5keV) is slowed down in a target and re-emitted as slow positrons.[2] Although, 80% of the beam is lost in a single re-moderation process, the brightness enhancement is still a factor 20 in each stage.

The final spot size of $\sim 1\mu\text{m}$ is achieved by three stages of brightness enhancement, each stage producing a compression of ~ 10 in diameter. Since we have 3 stages of reflection re-moderation, the microprobe can be compartmentalized into four straight sections. The angle between each section is chosen to optimize the overall layout of the microprobe (see Figure 1). Re-moderation using thin foils in a “straight-through” mode would allow a much simpler lens geometry, but we decided against such an arrangement for the lack of a reliable method to produce the necessary thin film re-moderators.

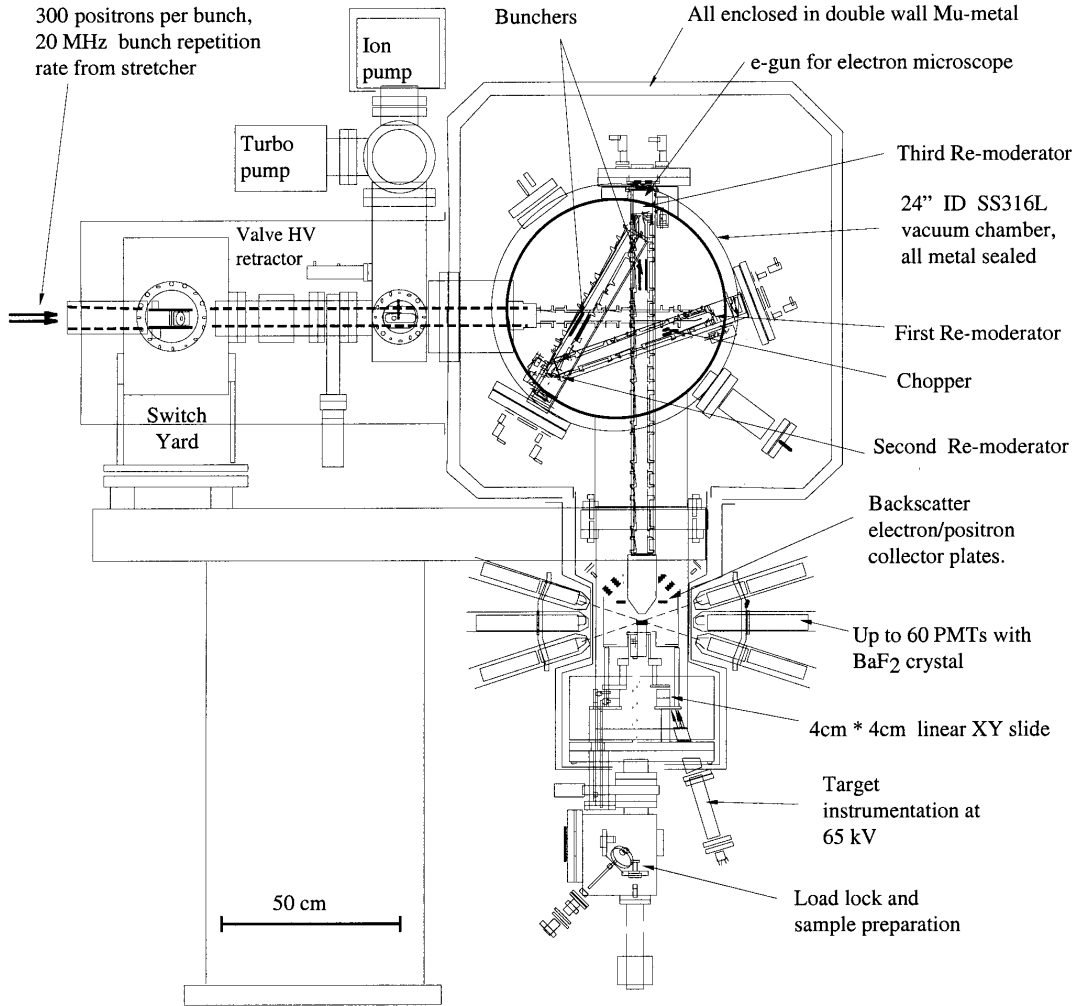


Fig 1. A side view of the LLNL positron microprobe. The vacuum system is completely enclosed in a double walled mu-metal shell.

The whole microprobe uses only electrostatic elements for focusing and steering, similar to the instrument built by K.F. Canter.[3] Magnetic systems would have too much crosstalk. All elements are designed using the ion simulation program SIMION. The first re-moderator is located 190 cm from the magnetic grid. The length of this column and the other three is chosen to reduce the background in the data acquisition window (see below).

The second column is 50 cm long and produces a spot of 0.15 mm on the second re-moderator. This column contains a chopper section, a set of parallel plates (4mm×4mm, 6mm separation) designed to let the positron bunch go through while deflecting the out-of-phase positrons leaking out of

the stretcher into a radiation shielded absorber. For 47 ns out of the 50 ns repetition cycle, there are about 3 Volt between the plates, and for a 2 ns duration the voltage is reset to zero with a 500 ps fall and rise time to let the positron bunch pass through. The buncher is located at an intermediate focus to minimize the jitter in the next beam spot caused by the imperfection in the chopper voltage.

The third column (50 cm long) contains the main buncher which will compress the time distribution of the beam from ~ 1 ns to 100 ps. The time trigger from the main buncher will be used to obtain the arrival time of the positron bunch at the target. The re-moderation process in the third re-moderator will add a negligible time spread to the timing. The third column produces a beam spot of 15

μm on the final re-moderator. The buncher operates with a tuned 30 V sine-wave at 80 MHz, using only every fourth phase.

The typical energy of the positrons in the chopper, buncher and final column is ~ 200 eV, with a 5 keV acceleration into the next re-moderator. Both the chopper and buncher column contain a 8-pole electrostatic deflector system to produce a round and centered beam spot on the next re-moderator. All three re-moderators are 3 mm diameter and 2 mm thick pure tungsten crystals, pre-annealed in an external tungsten oven. The crystals are placed behind an aperture made from a 25 μm thick SS foil with a hole diameter slightly larger than the anticipated beamspot. This allows tuning the positron beam onto the center of the optical columns and to cut halo positrons at a place where the timing of the annihilation does not cause unwanted background. The crystals can be annealed in-situ to a temperature above 2000 C by flipping them out of their normal position and in front of an electron gun heater.

The final column contains the elements that are necessary to focus the beam to a $\sim 1\mu\text{m}$ spot and to vary the beam energy from 1 keV to 50 keV. The final column (113 cm long) contains another buncher section that can be used to provide a tighter time distribution for specialized applications. If we use this second buncher, the beam spot size on the target will be limited to $\sim 7 \mu\text{m}$. This column also incorporates a 12-pole deflector lens system to steer the beam across the target. The 12-pole lens also can produce a line focus to scan with greater precision along one dimension. Half way between the final re-moderator and the target is a piezo-actuator driven collimator. The four independent baffle plates can collimate the beam at a location where the beam diameter is large. Using only the inner core of the beam, a much tighter beam spot can be achieved at the cost of intensity. This feature will be used to scan small areas of specific interest.

The influence of mechanical misalignments of the electrode structures, residual ac and dc magnetic fields, variations in the power supply voltages (ac ripple), and radiation background from successive stages of moderation were all checked and minimized. Since we add about 5000 Volt at every re-moderation stage, the re-moderators and their associated optical columns are an increasing high voltage. The HV power supplies and RF generators are floating at 5 kV, 10 kV and 15 kV respectively. The target voltage is between 16 kV and 65 kV relative to ground potential. The target instrumentation floats at up to 65 kV. All instruments used in the microprobe are fully computer controlled, we have about 100 different high voltage supplies in

four different main-frames.

5. The magnetic compensation

The lens elements are distributed to produce an odd number of beam crossovers in each column, and the cross-over positions are adjusted to compensate the beam spot shifts from residual ac magnetic fields. Therefore, a small magnetic side-field will not alter the focal-position in first order. The microprobe columns are housed in a double walled μ -metal enclosure, and the residual ac magnetic field is expected to be much less than 1 milli-Gauss. It is important to note that a small dc background field will only shift the final focus spot without degrading the spot size. The whole microprobe area is located inside a large 3-D Helmholtz coil, located around the edges of the room. The coils will lower the earth field to a few milli-Gauss in the microprobe area. The typical magnetic ac field in the area is less than a few milli-Gauss. The double walled mu-metal enclosure will cut this ac field to about 10 micro-Gauss. No electric current carrying instrumentation is located inside the mu-metal shield. The vacuum enclosure of the microprobe is made from SS316L with some SS304L, the lens elements are gold-plated aluminum, electric insulation and the lens carriers are alumina ceramic and radiation shielding is non-magnetic tungsten alloy.

6. The target

With 30 detector pairs in an opposing coincidence arrangement, the 10^7 positrons/s on the sample will give us a useful countrate of about 200 kHz, far more than any other existing positron lifetime spectrometer. The sample is located about 20 mm below the final focusing lens. For normal operation we plan to move the target for the X-Y scan and not sweep the beam. Moving the target keeps the beam-focus stationary and does not change the X-Y scale depending on positron energy. It also provides for a fixed focus, avoids non-linear X-Y corrections and does not change the time-of-flight for the positrons. It also does not change the backscatter geometry. The surface topography of the target has to be known to a few μm to correct for the limited focal-depth. A change in the surface elevation can also introduce a shift in the lifetime spectrum centroid by adding 1 ps positron time-of-flight for every 25 μm in target elevation change.

The typical measurement time for each 3-D image will be one or two days. Many different sample materials can be studied. For example, grain

boundaries can be resolved in 3-D or slip planes after stressing the material. Buried interfaces can be analyzed. Electro-migration of defects in integrated circuits can be investigated in-situ. We plan to study a wide variety of metals, plastics and composite materials. The development of radiation induced defects can be measured. With an instrumented target we will be able to heat the target in-situ to study the annealing behavior of many materials. We will also be able to cool or stress the sample.

We concluded that a magnetic focusing lens at the target is not practical for our positron microprobe. With a beam spot of only 1 μm all the backscattered positrons will follow a very tight path back up into the final accelerating lens, where they reflect, and return to the target within a few nano-seconds. Even the dramatic expansion of the magnetic field above the target does not expand the backscatter beam enough to be captured in a baffle. A deflector system is also of little help, since the backscatter beam overlaps with the incoming beam and contains similar positron energies. For this reason, we use electrostatic focusing only. This allows most of the low energy backscattered positrons to be harvested onto electrostatic collector plates located around the final lens, similar to the collector plates in a scanning electron microscope. An electron gun mounted behind the final re-moderator can convert the positron micro-probe into an electron microscope to study the surface in a classical way.

7. The time-of-flight background reduction

The optical column layout and their lengths are optimized to reduce the background radiation in the detector system. Since the positron bunches are spaced 50 ns apart, there are several bunches in the system at any given instant. The positron annihilations from the magnetic spider, accelerating grids, moderators, stopper for the chopped beam and collimators can all contribute to the background radiation. Since most of these elements are located past the first pre-buncher, their 511 keV radiation is also bunched and can distort the lifetime spectra in a very unpleasant way. The time-of-flight between all elements is chosen to produce all these annihilations in a 10 ns window before the target annihilations. We will have a clean data collection time window of 40 ns that is not disturbed by annihilations from other sources. Since the intensity of 511 keV radiation at the re-moderators is about 100 times higher than the target signal itself, this has been an important factor in the layout of various elements.

The location of the radio frequency elements

are optimized to reduce coupling effects. When the chopper triggers for the 2 ns long gate to let the positron bunch pass straight through, other bunches are located inside well-shielded electrostatic tubes.

Lifetime spectra will be collected using an array of BaF₂ detectors. The positron back-scattering from the target can cause an unwanted, time correlated background signal, and is a serious problem in many existing positron beam lifetime systems. We have designed our paired-coincidence detector system to record only events emanating from a small volume around the target. The 250 ps time resolution of the BaF₂ detectors is good enough to cut events which have an asymmetric time-of-flight for the 2 511 keV gamma rays. The enclosure around the target is large enough to discriminate events that originate from the wall.

Measuring both 511 keV arrival times independently, one can improve the overall time resolution by about 30%. The events from the detector array will be processed in a VME based TDC system using a "time-stretcher" technique with 25 ps resolution. Beside the time, we also record the energy signal from each event using the TDC's time-over-threshold feature. The expected data volume is phenomenal, we expect to process more than 10 MB of data per second in a PC-in-VME based system. The data are stored as a 128*128 matrix of 2k spectra, one spectrum for each X-Y pixel.

8. Conclusion

When completed, the LLNL positron microprobe will produce 3-dimensional images of the defect density in solid targets. Lifetime differences down to 1 ps can be extracted for single pixels, or 0.1 ps for larger areas. The positron microprobe will be a new high precision tool to study atomic scale defects.

Acknowledgements

We wish to thank D. Carter for excellent technical help, Drs. W. Trifhäuser, G. Kögel, P. Sperr, D.T. Suzuki, K.F. Canter and D.T. Britton for valuable discussions. This work was performed under the auspices of the US Department of Energy by LLNL under contract No. W-7405-ENG-48

REFERENCES

[1]. See for example, Proceedings of the 11th International Conference on Positron Annihilation, Eds, Y.C. Jean, M. Eldrup, D.M. Schrader, and R.N. West, Materials Science Forum, 255-257, Trans Tech Publications, USA (1997)

[2]. A.P. Mills, Jr., Appl. Phys., **23**, 189 (1980).

[3]. K.F. Canter, In Slow Positron Techniques For Solid Surfaces, AIP conference proceedings 303 (1994), p. 385